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(54) Title: TREATMENT OF CELLULOSIC MATERIAL AND COMPOSITIONS FOR USE IN THIS

(57) Abstract

Waste paper, especially office waste including laser and/or photocopier paper, is subjected to de-inking by pulping in the presence of a water-soluble anionic polymer at a substantially neutral pH and then by flotation in the presence of a cationic surfactant.

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Treatment of Cellulosic Material and Compositions for use in this

This invention relates to the de-inking of waste paper. In this specification we use the term "paper" to be generic to cellulosic sheet materials including filled and unfilled papers and paper board.

It is standard practice to reclaim waste paper to allow the reclaimed paper fibres to be used as part or all of the stock of subsequent paper production. The waste paper needs to be de-inked and it is standard practice to achieve this by forming a pulp from the waste paper in an aqueous medium that includes de-inking chemicals, and separating the ink from the pulp. This separation may be by, for instance, washing and/or flotation.

A typical de-inking process can comprise forming a pulp of the paper in the presence of the de-inking chemicals so as to disperse the ink into the pulp and then subjecting the resultant pulp to flotation to provide a Reject fraction that contains the floated ink components with a minimum of paper fibres, and an Accept fraction containing the paper fibres with a minimum of the ink components and the Accept fraction is then thickened to provide stock or other material that may be used for paper manufature, optionally after further conventinal treatments.

Typical chemical processes for de-inking are described in U.S. 1,925,372, 2,525,594, 4,347,099 and 4,780,179, and FR 2,281,456. As is apparent from these publications, typical de-inking chemicals have comprised a complex mixture of chemicals. Generally they include a significant amount of alkaline material, with sodium silicate, hydroxide and carbonate frequently being proposed.

The literature shows that the pH of the resultant pulp during the de-inking process is usually high, for instance above 9 and most usually above 10. Exposing the cellulosic fibres to this degree of alkalinity tends to

cause yellowing of the fibres and so it is generally necessary to add a peroxide as a bleaching agent.

It is also known to include various other additives.

The use of certain polycarboxylic acid polymers to activate de-inking processes is described in GB-A-2,178,079.

In U.S. 4,347,099 it is proposed to use a mixture of sodium hydroxide, hydrogen peroxide, a carboxylic polymer (usually polyacrylic acid or, preferably, poly hydroxy acrylic acid), dispersants such as anionic, cationic or non-ionic surface active agents, and other additives such as collectors, foaming agents, alkali silicates and pH regulators. The use of this complex and highly alkaline mixture is undesirable since the liquor that results from the de-inking process can cause environmental disposal problems. Neutral de-inking systems have the advantage that they reduce the risk of unwanted bleaching of the fibres and they have the advantage that they are more environmentally acceptable, especially because the effluent can be substantially neutral. Unfortunately such systems are not always as efficient as would be desired.

A de-inking system which has been commercialised under the trade name Ennesco D is described in WO90/10749 and apparently consists solely of a mixture of sodium carbonate and sodium phosphate, optionally with hydrogen peroxide. The use of this can reduce some environmental problems but it introduces others, since the presence of large amounts of dissolved phosphate in the de-inking liquor is environmentally undesirable. It is possible to operate this system at a lower pH, for instance around pH 8.

We have described in our application PCT/GB93/00780 (not published at the priority date) processes in which pulp is formed from the waste paper in an aqueous medium that has a pH of between 6 and 9 and that is substantially free of dissolved phosphate and that includes an ink dispersing amount of a dispersing agent which is preferably a polycarboxylic acid. It is stated that the pH is

preferably buffered to a value in the range 6.5 to 8 and the preferred dispersing agent is low molecular weight polyacrylic acid.

Various inks may be present in the paper that is to be de-inked and many of them comprise a binder and pigment. Many of the binders dissolve or soften sufficiently in the presence of the de-inking chemicals and/or during the pulping stage in order to release the pigment as a dispersion in the pulp. For instance many binders are alkali-soluble or alkali-swellable and the alkaline conditions prevailing in a conventional de-inking pulp will be sufficient to release the ink from the paper fibres and to release the pigment from the binder. As a result, the pulp has finely dispersed pigment throughout it and a test of a good de-inking process is the whiteness of a sheet made from the Accept fraction, optionally after washing it. The finely dispersed pigment has a tendency to cause an overall grey colouration if the de-inking process is inefficient.

However in addition to the problem of overall greyness, there is also a problem of visible specks in a sheet made from the Accept fraction. These specks arise when the ink particles on the paper that is to be de-inked are resistant to the de-inking chemicals and the pulping conditions as a result of the particles comprising a pigment and a binder that is sufficiently resistant to these conditions that ink particles retain their integrity and appear as specks in the Accept fraction (hereinafter referred to as a resistant binder). For instance a binder that is soluble or swellable at pH 9 sufficient to release pigment particles at that pH may not adequately release them when the pulping and de-inking process is conducted at a lower pH. Accordingly de-inking at relatively neutral pH values (for instance pH6 to pH8.5) can incur the disadvantage that ink binders that might release pigment during the pulping process in alkaline pulping are resistant during the neutral pulping such that they do not

release the pigment. Thus the ink particles may be resistant to dispersion of the pigment (hereinafter referred to as resistant ink particles) during neutral pulping even though they might swell and release the pigment during alkaline pulping.

A particular problem arises when the ink is applied by laser printing or by Xerographic or other photocopying techniques since ink deposits bonded by the binders used in these processes have a tendency to be released from the pulp but to remain as dispersed particles of bonded pigment. Binders that are chemically cross linked are liable to promote this problem. The ink particles can be relatively large and so have a severe tendency to remain trapped by the fibres in the Accept fraction in preference to being floated with any dispersed pigment in the Reject fraction. As a result, sheets made from the Accept fraction may have very good whiteness (as a result of good removal of any dispersed pigment by the de-inking process) but may have a significant number of visible specks (as a result of resistant ink particles remaining in the Accept fraction). Accordingly, neutral de-inking processes have advantages from an environmental point of view but have disadvantages in that binders that might swell in alkaline de-inking are resistant binders in neutral de-inking thereby leading to increased speck contamination, and speck contamination is additionally an increasing problem because of the increased amounts of laser or photocopier printed papers in paper waste.

There is therefore a need to find a way of improving neutral de-inking processes so as to reduce the speck contamination in the Accept fraction due to resistant binder, especially due to laser or photocopier inks, while maintaining good whiteness.

A process according to the invention for de-inking paper carrying ink particles comprising a binder, which is preferably resistant, and a pigment comprises forming a pulp of the paper and thereby dispersing the particles in

the pulp often substantially without release of the pigment from the particles, subjecting the pulp to flotation and thereby forming a Reject fraction containing the ink particles and an Accept fraction, and thickening the Accept fraction, and in this process the pulping is conducted at a pH of from 6 to 8.5 in the presence of an anionic polymer formed from water-soluble ethylenically unsaturated monomer or monomer blend comprising ethylenically unsaturated carboxylic acid and that promotes dispersion of the ink particles in the pulp, and the flotation is conducted in the presence of a cationic surfactant that promotes preferential flotation of the ink particles into the reject fraction.

Thus in the invention the flotation step is optimised by the use of cationic surfactant so as to obtain optimum flotation of the resistant ink particles and we generally find that it is possible to do this without adversely affecting the whiteness due to dispersion of pigment in the pulp.

In some instances it is desirable to subject the pulp to a plurality of flotation stages in which event one of the flotation stages may be optimised for removing dispersed pigment in the Reject fraction and another flotation stage may be optimised for removing resistant ink particles in the Reject fraction. For instance the surfactant may be added to the Accept fraction from a previous flotation stage so that dispersed pigment is primarily removed during the previous flotation stage and the resistant particles are primarily removed during the later flotation stage.

It is also possible to conduct a pulping and flotation de-inking process to remove dispersed ink and then to conduct pulping on the Accept fraction from the first process followed by flotation to remove resistant ink particles, or the process can be conducted the other way round.

The de-inking process of the invention is a substantially neutral de-inking process, that is to say the pulping is conducted at a pH which is substantially neutral, namely with a pH in the range of around 6 or 6.5 to 8 or 8.5. Often the pH is in the range of around 6.5 to 7.5 or 8.

The pH value is the pH of the pulp during the pulping process, i.e., after the inclusion of any de-inking additives that are incorporated in the pulp in order to promote de-inking. Alkaline pulping is conducted in the presence of materials such as sodium phosphate, sodium hydroxide and/or sodium silicate and often with peroxide as a bleach. Although small quantities of such materials can be included, provided the pH stays within the substantially neutral range, in the invention the pulping is preferably conducted in the substantial absence of such alkaline materials. Some bleaching chemicals can be included if necessary, or a subsequent bleach step can be added, but preferably the pulping is conducted in the substantial absence of peroxide or other bleaches.

Preferably the pulping is conducted in the presence of a buffer for providing the pulp with the desired pH, which is 6 to 8.5, often 6.5 to 8.

Although it is possible for the process to be conducted utilising sodium carbonate and sodium phosphate with the anionic polymer, preferably the process is conducted in the substantial absence of phosphate. Conventional buffers such as sodium carbonate and/or sodium bicarbonate, for instance as described in PCT/GB93/00780, are preferably used.

When buffer material or other material is being included to control or adjust the pH of the pulp, the proportions of buffer or other material and polymeric dispersing agent, on a dry weight basis, are generally 1:5 to 5:1 by weight, most preferably 2:1 to 1:2.

The anionic polymer that must be incorporated in the pulping composition may be an anionic dispersing agent or

an anionic anti-redeposition aid. It must be a water soluble polymer.

Its molecular weight (measured by gel permeation chromotography) should normally be above 1,000 and generally above 2,000. It is usually unnecessary for it to be above 200,000 or 150,000 and generally it is below 100,000. For instance it is often below 50,000. Best results are generally obtained when the molecular weight is in the range 2,000 to 20,000. However good results can also be obtained at higher values, e.g., 75,000 or higher.

If it is desired to provide the de-inking chemicals in solid form, it is convenient for the molecular weight to be sufficiently high to facilitate the formulation of the product as a solid, in which event the molecular weight is generally above 5,000 and frequently in the range 7,000 to 20,000. However when the de-inking chemicals are formulated as a solution the molecular weight can be lower. Best results are often obtained in the range 2,000 to 10,000, often around 2,500 to 6,000.

The monomeric material from which the polymeric dispersing agent is formed preferably comprises ethylenically unsaturated carboxylic acid. This can be methacrylic acid, maleic acid, crotonic acid, itaconic acid or any of the other polymerisable carboxylic acids, but preferably it is acrylic acid or a mixture of acrylic (or sometimes methacrylic) acid with maleic acid (frequently including anhydride). The acrylic acid or other carboxylic monomeric material can be polymerised alone or with sulphonic monomer such as 2-acrylamido methyl propane sulphonate (AMPS, U.S. trade mark), vinyl sulphonate or (meth) allyl sulphonate, and/or it can be copolymerised with non-ionic monomer, especially acrylamide.

Generally the carboxylic monomer constitutes at least 50% by weight of the monomers and preferably the polymer is formed from carboxylic monomer alone or a blend consisting of carboxylic and sulphonic monomers. Suitable polymers include polyacrylic acid, copolymers of this with maleic

anhydride, and copolymers of acrylic acid with 10-50% by weight AMPS.

The polymer preferably has polydispersity below 2, preferably below 1.8 and most preferably below 1.5, and generally above 1.1, for instance as described in EP 129,329.

Other dispersing agents can be included in the pulping process in order to improve results.

Suitable dispersing agents that can be used include cellulosic derivatives, polyphosphonates, bentonites, and sequestering agents.

Suitable cellulosic derivatives include cellulose ethers, such as methyl cellulose, and carboxy methyl cellulose.

Suitable bentonites include the various swelling clays that are referred to colloquially as bentonites, including true bentonite, Fuller's Earth, hectorite and various swelling montmorillonites, such as activated calcium montmorillonite.

Suitable sequestering agents are amino carboxylic acid sequestering agents, such as ethylene diamine tetro acetic acid and nitrilo tri acetic acid.

Suitable mixtures of two or more of the dispersing agents include mixtures of one of the described polymeric dispersing agents, especially polyacrylic acid or a copolymer of acrylic acid with maleic acid (including anhydride) or AMPS, with a polyphosphonate or sequestering agent or CMC or methyl cellulose.

The components in such mixtures typically are present in proportions ranging from 1:3 to 3:1 by weight.

The amount of dispersing agent that is required for optimum performance can be selected by routine experimentation and is normally in the range 0.01 to 1% by weight based on the dry weight of the pulp, generally 0.05 to 0.5%.

The cationic surfactant is preferably added to the pulp after the pulping process so as to promote

preferential flotation in the pulp. However it is also possible for some or all of the cationic surfactant to be included in the pulping process. For instance it may be included initially in the water into which the waste paper is pulped or it may be added with the polycarboxylic acid and any other de-inking chemicals during the pulping process.

The cationic surfactant that is used in the invention can either be a true cationic surfactant (in the sense that it is cationically charged at the time it is added to the pulp) or it can be a free base surfactant which we believe can become cationically charged by interaction with the polycarboxylic or other dispersing agent or other component of the pulp.

Suitable materials that can be used include ethoxylated fatty amines and fatty diamines and quaternary fatty ammonium compounds (i.e., quaternary ammonium compounds including at least one fatty group). The fatty groups can be naturally occurring or synthetic fatty groups, generally containing 6 to 24, often 8 to 18, carbon atoms. The fatty group is often alkyl. Ethoxylate fatty amines are preferred.

The cationic surfactant can be a surfactant that includes both cationic and anionic groups (i.e., amphoteric) but preferably it is wholly cationic.

The formation of the pulp may be conducted in the presence of additional materials but a particular advantage of the invention is that the chemicals used for it can consist essentially only of the dispersing agent, buffering alkali, optional surfactant and optional flotation aids and collectors. Peroxide or other bleach can be included if desired, but an advantage of the invention is that it is usually unnecessary. Accordingly the liquor resulting from the de-inking is relatively free of materials that would create environmental problems during disposal.

The de-inking process can be carried out in broadly conventional manner, except for the choice of the de-inking

chemicals, as described above. The de-inking chemicals can be included in the aqueous liquor into which the waste paper is initially pulped, or the waste paper can be pulped to form an aqueous pulp into which the de-inking chemicals are then incorporated.

The waste paper preferably includes photocopy (xerographic) and/or laser ink printed paper, for instance in an amount of 10 to 100%, often 50 to 100%, of the waste paper. The process is of particular value for de-inking office waste.

The overall de-inking process generally comprises a series of stages, typically consisting of an initial maceration or pulping stage (preferably conducted under very high shear), a screening stage to remove grit and oversized particles, one or more flotation stages, and a thickening stage to form a clean pulp that can either be used as such or that may be drained to form a dried pulp. Typical de-inking processes are described in more detail in, for instance, Handbook for Pulp and Paper Technologists by G.A.Smook.

Thickening can be conducted on thickening drums in conventional manner, for instance to raise the solids content of the pulp from a concentration suitable for flotation to a concentration suitable for recycling to paper manufacture, for instance 5 to 15% dry weight. The Accept fraction from flotation may be subjected to washing with water or an aqueous solution of surfactant before or after thickening.

The following are examples of the invention. In these examples the following de-inking components are used.

A	50:50 mix of sodium polyacrylate molecular weight around 5,000 and sodium carbonate
D	Ethoxylate fatty amine cationic surfactant
E	C12 alkyl quaternary ammonium salt catinic surfactant
F	Amphoteric cationic surfactant
G	Sodium lauryl sulphate anionic surfactant
H	Sodium dioctyl sulpho succinate non-ionic surfactant

Example 1

A series of de-inking processes was conducted. In each process 1.5kg of waste paper carrying either laser print or Xerox (trade mark) print was pulped with 0.2% (based on the dry weight of waste) of de-inking component A and 10 litres of water under neutral conditions. The pulp was diluted to 1% and a sample was taken for speck analysis. The results of this analysis are quoted in column of Table 1.

In this, and in all instances where a sample was taken for speck analysis, the sample was made into a handsheet, dried and a visual speck count of the dried handsheet was made. The results were assessed on a scale of 1 to 10 where 10 is the worst (highest number of specks) and 1 is the best (lowest number of specks).

In those instances where surfactant was to be added, it was then added to the pulp in an amount of 0.125%. The pulp, with or without surfactant, was then floated in a laboratory Voith flotation cell for 2 minutes. The Reject was scraped off the surface and the Accept after 10 minutes was sampled. Speck counts were made on each sample and the results are in column 2 of Table 1.

The 1% Accept after 10 minutes was thickened to 10% over a 710μm sieve and the thickened stock was sampled and a speck count was made. The results are in column 3 of Table 1.

Table 1

Additives	Laser			Xerox		
	1	2	3	1	2	3
Blank	10	9	9	10	9	9
Soap	10	5	4	10	9	9
A	10	9	8	10	8	8
A + D	10	3	2	10	4	4
A + E	10	3	2	10	3	3
A + F	10	5	4	10	6	6
A + G	10	4	4	10	6	6
A + H	10	6	5	10	7	7

It is apparent from these results that the cationic surfactants D and E give particularly good results.

Example 2

Laser printed paper and xerographic printed paper was pulped with 0.2% of de-inking component A at neutral conditions (around pH7.4 or 7.5), diluted, treated with surfactant, and subjected to flotation all broadly as described in Example 1. The dirt area of the hand sheet was recorded in parts per million by image analysis and the brightness was recorded. The results are set out in Table 2, in which columns 1, 2 and 3 have the same meanings as in Example 1.

Table 2

	Dirt Area			Brighteners		
Laser	1	2	3	1	2	3
A	20354	6618	4887	73.5	75.3	76.2
A + D	10467	80.5	96.6	74.2	76.7	79.9
A + E	14235	475	733	73.2	76.1	79.3
A + F	10193	274	258	72.7	75.4	80.8
A + G	22560	2222	902	69.2	73.3	76.4
A + H	11192	628	306	71.7	74.0	78.6
Xero-graphic						
A	10129	10290	10411	74.4	74.4	75.1
A + D	9525	1087	483	73.1	75.4	79.4
A + E	8470	523	668	75.4	75.4	77.6
A + F	9058	684	854	76.2	76.8	80.7
A + G	11812	1876	1957	73.9	78.4	76.8
A + H	11304	1232	790	73.2	74.9	78.9

These results demonstrate the low speck area and the satisfactory brightness values that can be obtained using the cationic surfactants. The amphoteric cationic surfactant gives a low speck value on the laser paper but is not so efficient on the xerographic paper.

The results also demonstrate the good brightness values obtainable in the invention, and in this respect the amphoteric cationic surfactant gives good results.

In this test the non-ionic surfactant H also gives reasonably good dirt area and brightness values, but not as good as the best cationic surfactant. However in the preceding example the non-ionic surfactant gave less satisfactory results.

The data shows that the use of the cationic surfactant does give valuable results on a variety of papers, especially on laser and xerographic papers.

According to another aspect of the invention, the pulping is conducted at a pH of 6 to 8.5 in the presence of an anionic polymer as desribed above, and non-ionic surfactant is then added to the pulp in order to promote preferential flotation of the resistant ink particles into the reject fraction. In this aspect of the invention, the cationic surfactant can be omitted.

In another aspect of the invention, the pulping is conducted in the presence of an anionic polymer as described above and the flotation is conducted in the presence of a cationic or non-ionic surfactant that promotes preferential flotation of the resistant ink particles into the reject fraction, but in this process the pulping is conducted at an alkaline pH, for instance up to 9 or 9.5. Thus the pulping can be conducted in the presence of an alkali, optionally with a bleach. The alkali is typically selected from sodium silicate and sodium hydroxide and sodium phosphate. The bleach is typically hydrogen peroxide.

In another aspect of the invention, the pulping is conducted at a pH of 6 to 8.5 as described above and the flotation is conducted in the presence of a cationic or non-ionic surfactant as described above, and the pulping is conducted in the presence of a blend of sodium carbonate and sodium phosphate instead of or in addition to the anionic polymer.

CLAIMS

1. A process of de-inking paper carrying ink particles comprising a resistant binder in the pigment, the process comprising forming a pulp of the paper and thereby dispersing the ink particles in the pulp substantially without release of pigment from the particles, subjecting the pulp to flotation and thereby forming a Reject fraction containing the ink particles and an Accept fraction, and thickening the Accept fraction, characterised in that the pulping is conducted at a pH of 6 to 8.5 in the presence of an anionic polymer formed from water soluble ethylenically unsaturated monomer or monomer blend comprising an ethylenically unsaturated carboxylic acid and that promotes dispersion of the ink particles, and the flotation is conducted in the presence of a cationic surfactant that promotes preferential flotation of the ink particles into the Rejection fraction.

2. A process according to claim 1 in which the pulp is formed and the cationic surfactant is then added to the pulp.

3. A process according to claim 1 or claim 2 in which the cationic surfactant is selected from amphoteric surfactants, surfactants which are cationically charged before addition to the pulp, and surfactants that are in the form of a free base that can become cationically charged after addition to the pulp.

4. A process according to claim 1 or claim 2 in which the surfactant is selected from ethoxylated fatty amines, fatty diamines, and quaternary fatty ammonium compounds.

5. A process according to claim 1 or claim 2 in which the surfactant is an ethoxylated fatty amine.

6. A process according to any preceding claim in which the pulp has a pH of 6.5 to 8.

7. A process according to any preceding claim in which the pulping is conducted in the presence of a buffer for providing the pH of the pulp.

8. A process according to claim 7 in which the buffer comprises sodium carbonate and/or sodium bicarbonate.

9. A process according to any preceding claim in which the anionic polymer is a water soluble polymer having molecular weight 2,000 to 100,000 and formed from monomer of which at least 50% by weight is ethylenically unsaturated carboxylic acid.

10. A process according to claim 9 in which the dispersing agent is a polymer of a carboxylic acid monomer selected from acrylic acid, methacrylic acid, maleic acid, crotonic acid and itaconic acid.

11. A process according to any preceding claim in which the anionic polymer is a polymer of acrylic acid having molecular weight 2,000 to 20,000.

12. A process according to any preceding claim in which the waste paper comprises office waste including laser printed paper and/or photocopier paper.

13. A process according to any preceding claim in which the pulping is conducted in the substantial absence of added alkali or bleach.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 94/02350

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 D21C5/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,A	WO,A,93 21376 (ALLIED COLLOIDS) 28 October 1993 cited in the application see claims 1-16 ---	1-13
A	WO,A,93 02967 (ALLIED COLLOIDS) 18 February 1993 see claims 1-12 ---	1-13
A	WO,A,93 02966 (ALLIED COLLOIDS) 18 February 1993 see claims 1-13 -----	1-13

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report
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Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
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